



## REVIEW ARTICLE

# Fabricating Ultrafine-Grained Materials through the Application of Severe Plastic Deformation: a Review of Developments in Brazil

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Considerable attention is now being devoted to the fabrication and properties of ultrafine-grained materials processed through the application of severe plastic deformation. The two main processing techniques for SPD are equal-channel angular pressing and high-pressure torsion. Ten years ago, in 2002, the first Brazilian research paper was published describing the results obtained from a material processed using an SPD technique. Since that time, Brazilian materials scientists have made, and are continuing to make, major contributions to this important field. This tenth anniversary, and the introduction of a new Brazilian research journal, provides an excellent opportunity to summarize the main principles of SPD processing and then to review the major contributions from Brazil in the field of SPD.

**KEY WORDS:** Equal-channel angular pressing; High-pressure torsion; Nanostructured materials; Severe plastic deformation; Ultrafine grain sizes

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## 1. Introduction

The processing of metals through the application of severe plastic deformation (SPD) has become important in materials research over the last twenty years. This interest has arisen because SPD processing provides an opportunity for refining the grains of conventional bulk solids to produce grain sizes within the submicrometer (100 nm–1.0  $\mu\text{m}$ ) or even the nanometer (<100 nm) range. Since many of the fundamental characteristics of polycrystalline materials are dependent upon the grain size, SPD processing has the capability of producing materials having unusual and attractive properties. For example, it is anticipated that materials having very small grain sizes will exhibit high strength and, if these ultrafine grains are reasonably stable at elevated temperatures, it should be possible to achieve an excellent superplastic forming capability.

A detailed review was published several years ago describing the basic principles of SPD processing<sup>[1]</sup> and more recently a report summarized the terminology associated with SPD<sup>[2]</sup>. Currently, it is now recognized that, although several procedures are available for applying SPD in metallic systems, there are two processing procedures that represent the major methods for achieving grain refinement. These two procedures are equal-channel angular pressing (ECAP)<sup>[3]</sup> and high-pressure torsion (HPT)<sup>[4]</sup>. In this report, sections 2 and 3 summarize the basic principles of ECAP and HPT, respectively. Materials scientists in Brazil have played an important role in establishing and conducting research on materials processed by SPD and the year of 2012 represents the tenth anniversary of the first Brazilian publication describing materials processed by SPD. Accordingly, these developments are presented in sections 4 and 5 that describe some of the current research that is now underway in the field of SPD processing in Brazil.

## 2. Processing by ECAP

Processing by ECAP refers to the situation in which a sample is pressed through a die constrained within an internal channel that is bent abruptly through a sharp angle. An example of the ECAP process is shown in Fig. 1, where the channel angle is  $90^\circ$ <sup>[5]</sup>. It is readily apparent that the sample emerges from the die having the same cross-sectional area as in the initial condition and it means that repetitive pressings can be conducted in order to impose very high strains.

The planes X, Y, and Z in Fig. 1 represent the transverse, flow, and longitudinal planes within the as-pressed billet, respectively.

A critical question in ECAP concerns the magnitude of the strain imposed in each separate pass through the die. In practice, it can be shown that this strain is dependent upon the angle between the two parts of the channel,  $\Phi$  ( $\Phi = 90^\circ$  in Fig. 1), and the angle representing the outer arc of curvature where the two parts of the channel intersect,  $\Psi$  ( $\Psi = 0^\circ$  in Fig. 1 since no curvature is indicated). A relationship was derived for the strain in each pass<sup>[6]</sup> and this relationship can be plotted as shown in Fig. 2 to provide a direct representation of the imposed strain in ECAP<sup>[7]</sup>. In Fig. 2, the strain  $\epsilon_1$  is shown as a function of the channel angle,  $\Phi$ , for the condition where the number of passes,  $N$ , is 1. It can be seen that the arc of curvature has only a minor effect on the total strain; and in practice, for a conventional die with  $\Phi = 90^\circ$ , the strain is approximately 1 on each separate pass.

The nature of the microstructure introduced by pressing through the ECAP die is dependent upon the processing route employed in conducting consecutive passes. Four distinct processing routes have been identified: in route A the

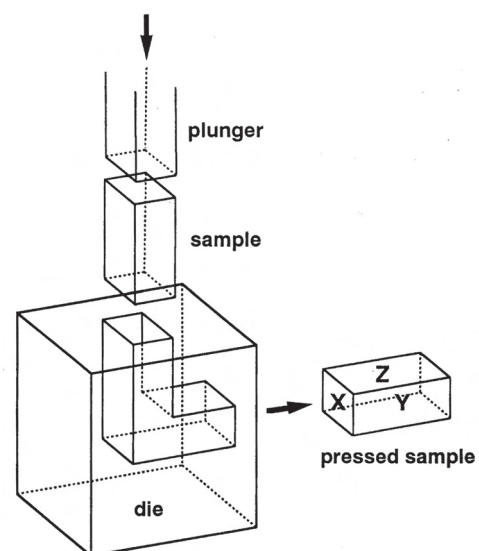


Fig. 1 Principle of the ECAP process using a die with a channel angle of  $90^\circ$ <sup>[5]</sup>

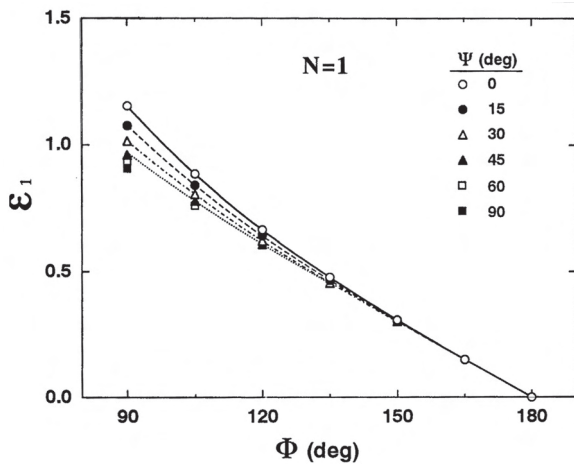


Fig. 2 The strain imposed in one pass of ECAP as a function of the channel angle,  $\Phi$ <sup>[7]</sup>

billet is removed from the exit channel of the die and then re-inserted into the entrance channel without rotation; in route B<sub>A</sub> the billet is rotated through 90° in alternate directions between each pass; in route B<sub>C</sub> the billet is rotated by 90° in the same direction between each pass; and in route C the billet is rotated by 180° between passes<sup>[8]</sup>. These different processing routes are important because they produce different slip systems within the billet. This is illustrated schematically in Fig. 3, where the X, Y, and Z planes correspond to the planes illustrated in Fig. 1 and the slip system is shown for each pass<sup>[9]</sup>. For example, in route C a rotation of the billet by 180° means that the slip in the second pass is on the same plane, but in the opposite direction, to the slip in the first pass and thereafter there is an alternation between these two slip directions. This means that route C is a redundant strain process and the same conclusion applies also to route B<sub>C</sub>, except that in the latter the first and third passes are in opposite directions and the second and fourth passes are similarly in an opposite sense. It follows from an analysis of these slip systems, and it can be shown experimentally with fcc metals such as aluminum<sup>[10]</sup>, that processing by route B<sub>C</sub> is the optimum condition in order to achieve an equiaxed grain structure with grain boundaries having high angles of disorientation.

When a metal is processed by ECAP, the grain size is generally significantly reduced and this should lead to a major

strengthening. An example of this effect is shown in Fig. 4, where experimental data are plotted for a series of commercial aluminum-based alloys<sup>[11]</sup>. For each alloy, the left axis denotes the 0.2% proof stress in the absence of ECAP and the points at equivalent strains of 1 and 2 represent the measured proof stresses after 1 and 2 passes through a die having a channel angle of  $\Phi = 90^\circ$ . Thus, all alloys significantly strengthen after one pass but thereafter there is only a very minor additional increase in strength.

Metals processed by ECAP should also exhibit good superplastic properties when tested in tension at elevated temperatures. An example of an exceptional superplastic elongation is shown in Fig. 5 for a magnesium ZK60 alloy containing 5.5% Zn and 0.5% Zr<sup>[12]</sup>. This alloy was extruded prior to ECAP in order to introduce a smaller grain size<sup>[13]</sup> and then pressed at 473 K, cut into a tensile specimen with the gauge length oriented along the pressing direction, and pulled in tension to failure at a temperature of 473 K using an initial strain rate,  $\dot{\epsilon}$ , of  $1.0 \times 10^{-4} \text{ s}^{-1}$ . The result shows excellent superplasticity with an elongation to failure,  $\Delta L/L_0$ , of 3,050%, where  $\Delta L$  is the change in length and  $L_0$  is the initial length, respectively. This is the highest tensile elongation recorded to date for any magnesium alloy processed under any conditions and also it is the highest superplastic elongation recorded in any metal after processing by ECAP. The absence of any necking within the specimen gauge length is conclusive proof for the occurrence of true superplastic flow<sup>[14]</sup>.

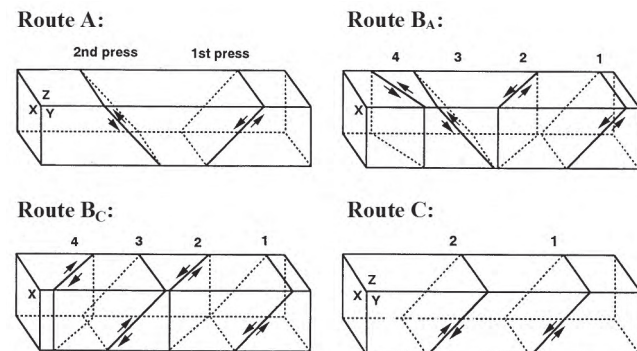


Fig. 3 The slip systems associated with the four processing routes in ECAP<sup>[9]</sup>

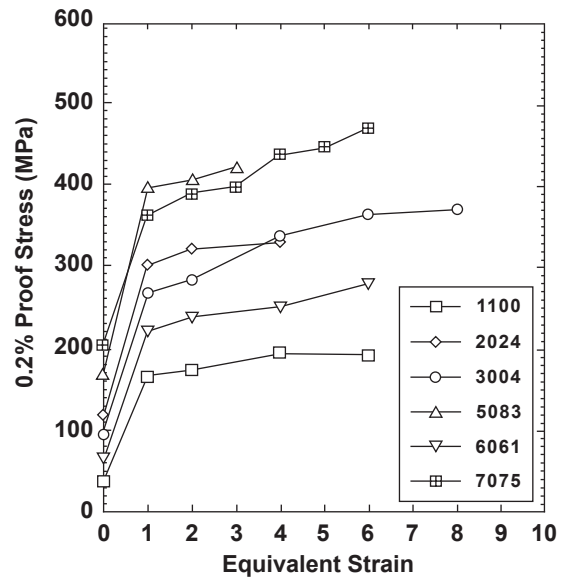


Fig. 4 Strengthening in commercial aluminum alloys processed by ECAP<sup>[11]</sup>

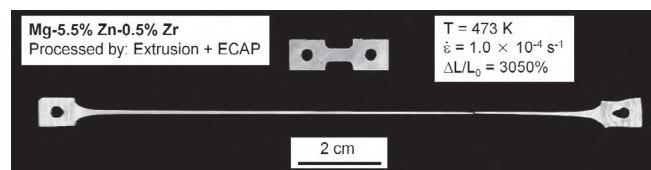


Fig. 5 Exceptional superplasticity in a magnesium ZK60 alloy processed by ECAP<sup>[12]</sup>

### 3. Processing by HPT

Processing by HPT is generally conducted using a thin disk which is placed between massive anvils, subjected to a pressure  $P$ , and then torsionally strained through rotation of one of the anvils: this situation is illustrated schematically in Fig. 6<sup>[15]</sup>. Some limited experimental results are available for small cylindrical samples processed by HPT<sup>[16,17]</sup> but most of the available data relate to the use of thin disks.

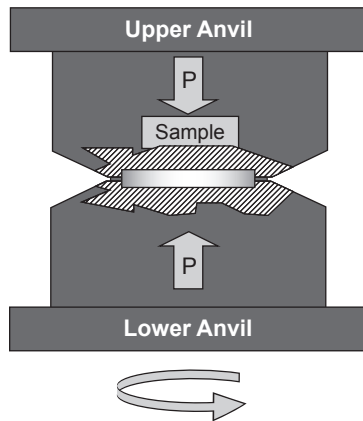


Fig. 6 Principle of processing by HPT<sup>[15]</sup>

A potential problem in HPT processing is that the strain varies across the disk with a zero strain at the disk center and a maximum strain at the periphery. This suggests that the structures produced by HPT processing may be inherently inhomogeneous but early experiments showed that a reasonable level of homogeneity may be attained across the disk's surface provided there was a sufficiently high applied pressure and the torsional strain was continued through a sufficiently large number of revolutions<sup>[18]</sup>. This structural evolution towards homogeneity has been effectively interpreted using strain gradient plasticity modeling<sup>[19]</sup>.

Very recently, interest has centered on whether there is homogeneity in the through-thickness of HPT disks parallel to the rotation axis. The experimental results shown in Fig. 7 are for high-purity (99.99%) aluminum tested by HPT through 1/4 turn (upper row) and 1/2 turn (lower row) using a pressure of 6.0 GPa at room temperature (RT)<sup>[20]</sup>. Three disks were used for each testing condition with one disk sectioned horizontally through the mid-plane to give the Center position and the other two disks polished inwards from the top and bottom surfaces by  $\sim 200 \mu\text{m}$  to give the Upper and Lower positions, respectively. Measurements of the Vickers microhardness,  $H_v$ , were recorded on the sectional planes of each of these three samples and the results shown in Fig. 7 use colors to designate different hardness values as represented at the lower right. The important feature of these results is that all planes of sectioning show

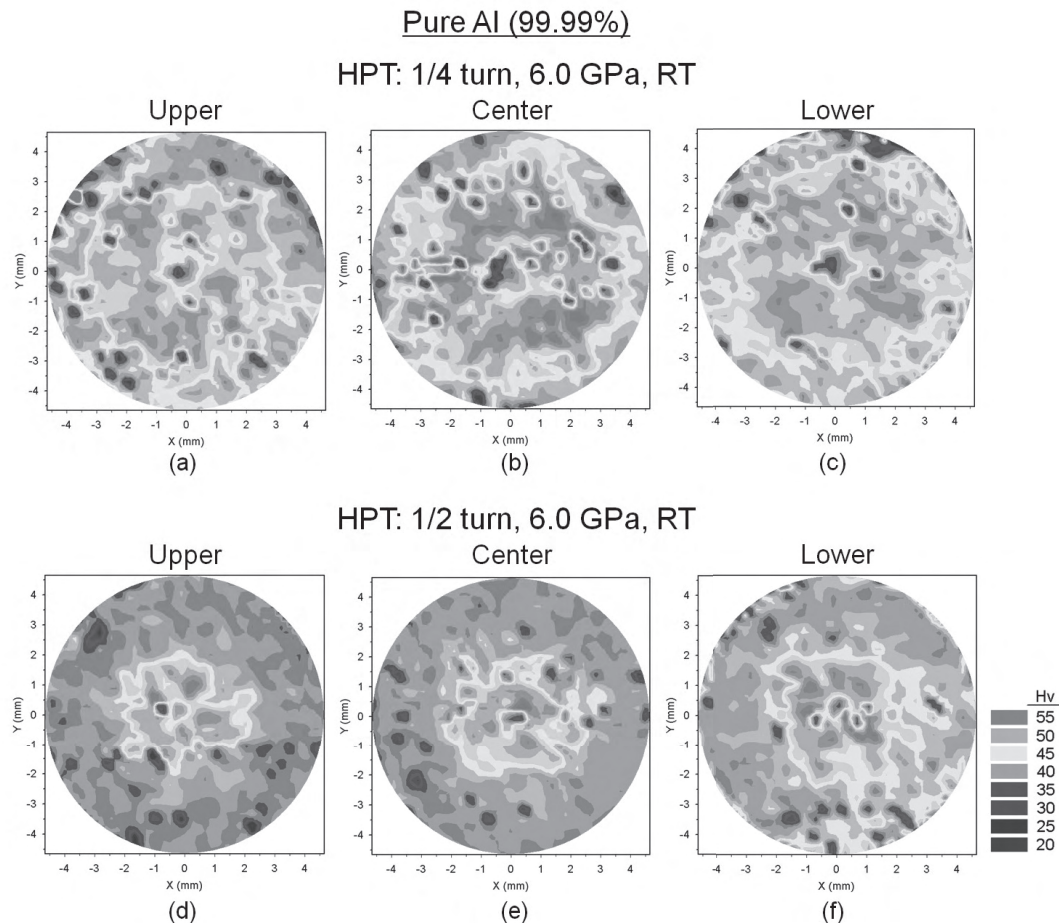


Fig. 7 Color-coded maps showing the hardness values recorded in high-purity aluminum after 1/4 turn (upper row) and 1/2 turn (lower row) of HPT in the center of the disk and near the upper and lower surfaces<sup>[20]</sup>

essentially the same result thereby demonstrating the presence of excellent homogeneity in pure aluminum after processing by HPT. It should be noted that these results contrast with magnesium alloys where there is evidence for significant inhomogeneities in the through-thickness after processing by HPT<sup>[21]</sup>.

As with samples processed by ECAP, processing by HPT also provides an opportunity for achieving significant superplastic elongations after torsionally straining. An example is shown in Fig. 8 for the Zn-22% Al eutectoid alloy<sup>[22]</sup>. For this material, the HPT was conducted at room temperature under an imposed pressure of 6.0 GPa at a rotation speed of 1 rpm and thereafter the disks were cut into miniature tensile specimens and tested in tension to failure using a strain rate of  $1.0 \times 10^{-1} \text{ s}^{-1}$  at a temperature of 473 K. The samples in Fig. 8 represent the untested condition (upper) and then processing by HPT through 1, 3, and 5 turns, respectively. It is apparent that the elongations to failure increase with increasing strain and the maximum elongation to failure of 1,800% for the sample processed through  $N = 5$  turns represents a record elongation for any material processed by HPT.

#### 4. Historical Developments in Severe Plastic Deformation in Brazil

The present paper marks the 10<sup>th</sup> anniversary of the first publication by a Brazilian research group where a material was processed using an SPD technique. High-Pressure Torsion was used to consolidate metallic powder and the results were reported in *Scripta Materialia* in 2002<sup>[23]</sup>. Within the last ten years, many papers have been published in the area of SPD by different Brazilian research groups covering the areas of the processing, structure, and properties of ultrafine-grained materials. In this research, different SPD techniques were employed including HPT, ECAP and Accumulative Roll Bonding (ARB) for the processing of the materials.

Initially, studies on SPD processing in Brazil focused on the ECAP technique. Finite Element Modeling (FEM) was used to determine the effect of processing parameters on the occurrence of billet cracking<sup>[24,25]</sup>, on the plastic flow<sup>[26,27]</sup> and on the homogeneity of the distribution of deformation<sup>[28,29]</sup>. The upper-bond theory was used to estimate the effect of diverse processing parameters on the amount of strain imposed during ECAP and the punch pressure developed during the process<sup>[30]</sup>.

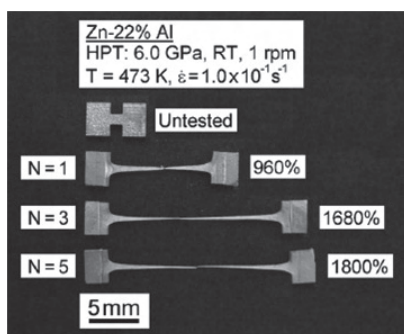


Fig. 8 Superplasticity in the Zn-22% Al eutectoid alloy after processing by HPT<sup>[22]</sup>

It should be noted that the research carried out in Brazil has strongly influenced the topic of ECAP processing. The occurrence of plastic instability, flow localization, segmentation and billet cracking was reported in several early papers on SPD published in the United States<sup>[31-33]</sup>. It was observed in this early work that flow softening materials tend to exhibit plastic instability during ECAP processing<sup>[31]</sup> and some difficult-to-work materials exhibit segmentation and cracking when processing at low temperatures<sup>[32,33]</sup>. These early publications stimulated the development of FEM in Brazil in order to model the occurrence of plastic instability<sup>[26,27]</sup> in flow softening materials. Fig. 9 shows the appearance of billets of flow softening materials after processing by ECAP<sup>[27]</sup>. Flow localization is clearly observed in the upper two billets but it was shown that increasing strain rate sensitivity could stabilize the plastic flow in this kind of material so that flow localization is not observed in the two lower billets. The occurrence of billet segmentation and cracking was also evaluated. It was shown that increasing the ECAP die angle is an effective way to process difficult-to-work alloys<sup>[24]</sup> and, in addition, the use of a back-pressure reduces the amount of damage introduced in the material during ECAP<sup>[27]</sup>.

Several different metallic alloys have been processed in Brazil using various SPD techniques including aluminum<sup>[23,25,29,34-40]</sup>, lead alloys<sup>[41,42]</sup>, magnesium<sup>[20,43]</sup>, and steel<sup>[44,45]</sup>. Ultrafine-grained structures, with grain sizes between 100 nm and 1,000 nm, were successfully intro-

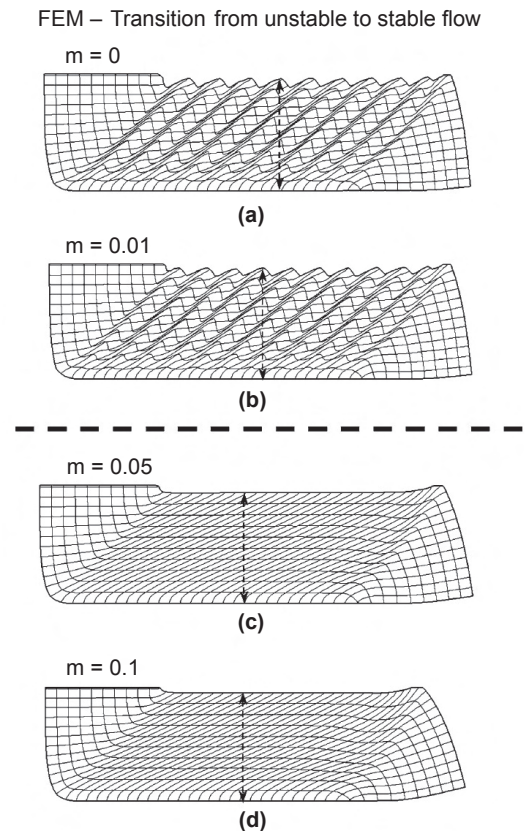
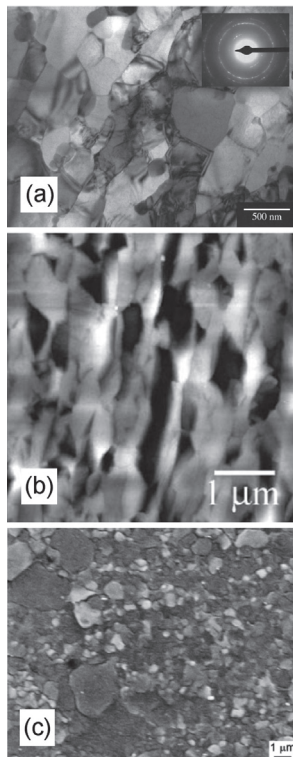


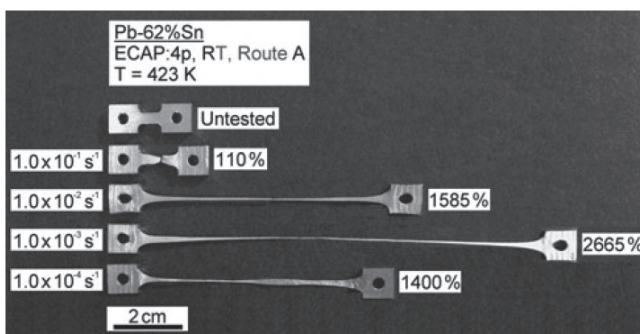
Fig. 9 Appearance of a rectangular grid pattern after simulation of ECAP considering different values for strain-rate sensitivity,  $m$ : the distortions of the grids delineate a transition from unstable flow at strain-rate sensitivities of (a) 0 and (b) 0.01 to stable flow at strain rate sensitivities of (c) 0.05 and (d) 0.1<sup>[27]</sup>

duced into all of these materials. Fig. 10 shows examples of such structures in (a) an aluminum alloy processed by ECAP through four passes<sup>[37]</sup>; (b) an IF-steel processed by ARB through three passes<sup>[44]</sup>; and (c) a magnesium alloy processed by HPT through one turn<sup>[20]</sup>.

It was observed that there is a general trend of increasing strength when processing aluminum<sup>[23,25,34,36-40]</sup>, magnesium<sup>[20]</sup>, and steel<sup>[44,45]</sup> by SPD. However, there is a decrease in strength when processing a lead alloy<sup>[41]</sup>. Superplastic ductilities were obtained in a lead-tin alloy after processing by ECAP and Fig. 11 shows the appearance of tensile samples of this alloy after pulling to failure at different strain rates at 423 K<sup>[42]</sup>.



**Fig. 10** Ultrafine-grained structures introduced in (a) an aluminum alloy processed by four passes of ECAP<sup>[37]</sup>; (b) an IF-steel processed by three passes of ARB<sup>[44]</sup>; and (c) a magnesium alloy processed by one turn of HPT<sup>[20]</sup>. The grain structure was evaluated by (a) transmission electron microscopy, (b) atomic force microscopy and (c) scanning electron microscopy.



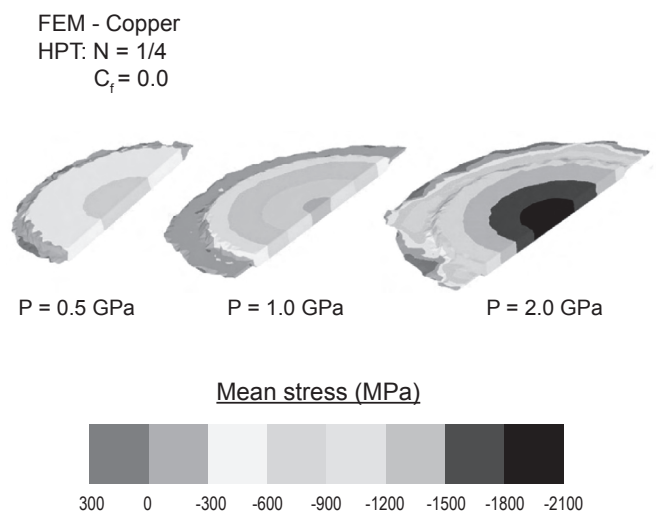
**Fig. 11** Appearance of tensile samples of the Pb-62% Sn alloy processed by ECAP and pulled to failure at 423 K at different strain-rates<sup>[42]</sup>

## 5. Current Trends in SPD Processing in Brazil

Following the general trend of the materials science community of increasing interest in processing by HPT, much of the research in Brazil is now focused on the various processing parameters associated with this technique. Recent reports have described the mechanisms of plastic flow during this process<sup>[46-48]</sup>. It was reported that the general belief that the hydrostatic pressure is constant in this process is not accurate. In fact, it can be shown by modeling that larger hydrostatic pressures develop in the center of the disc and a linear reduction is observed towards the edge of the disc<sup>[46]</sup>. Fig. 12 shows the distribution of the mean stresses in HPT predicted by FEM<sup>[46]</sup>. This result has implications for the evolution of the structure of materials processed by HPT which depend upon the hydrostatic pressure. It was also shown that the temperature rise may reach tens of degrees in HPT during the processing of hard materials and/or at high rotation rates<sup>[47]</sup>. Heterogeneities in structure and hardness distribution were observed along the sample thickness<sup>[49]</sup> and the sources of these heterogeneities are currently under investigation<sup>[48]</sup>.

The interest in the mechanical properties of materials processed by SPD is now significantly focused on practical applications. Knowledge of successful processing routes for difficult-to-work materials can be used to produce interesting combinations of properties in these materials. Thus, the processing of magnesium alloys by SPD has attracted significant attention from the scientific community due to the potential improvement of the mechanical properties of these materials with an overall objective of increasing their use in the transportation industry.

Many researchers are also interested in using SPD for processing biomaterials for use as biomedical implants. Early reports suggested that titanium could be processed only at high temperatures due to the occurrence of segmentation and cracking. However, the development of processing routes using ECAP dies with larger angles between the channels has permitted the processing of this material at room temperature. Recent publications report



**Fig. 12** Distribution of mean stresses during HPT considering different nominal hydrostatic stresses<sup>[46]</sup>

significant improvements in the mechanical properties of titanium processed by ECAP and promotes this material as a potential substitute for other alloys<sup>[50,51]</sup>. Table 1 shows the evolution of strength in titanium of grade II after processing by ECAP<sup>[50]</sup>.

There is also a current interest in Brazil in producing magnesium alloys with small grain sizes in order to promote grain boundary diffusion and thereby improve the efficiency of magnesium as a hydrogen storage material<sup>[52,53]</sup>.

Fundamental research is also of great interest in the SPD area. Recent experiments on copper<sup>[54]</sup> demonstrate that SPD processing may lead to an excess of vacancies in the material structure. This finding has great implications for research in materials science and it is reasonable to anticipate it will be the subject of extensive future studies.

**Table 1** Evolution of hardness in commercial purity titanium during ECAP processing<sup>[50]</sup>

Condition	Nominal equivalent strain	Hardness (Hv)
Coarse-grained	-	145
1 pass of ECAP	0.66	205
2 passes of ECAP	1.32	208
3 passes of ECAP	1.98	213
4 passes of ECAP	2.64	269

## 6. Summary and Conclusions

The processing of ultrafine-grained metals through the application of severe plastic deformation is a major research topic in modern materials science. The main processing techniques at the present time are equal-channel angular pressing (ECAP) and high-pressure torsion (HPT).

Starting 10 years ago, in 2002, with research on a material processed by HPT, there has been a continuous and significant contribution to this research field by materials scientists in Brazil. This research is now continuing with major emphasis in areas such as finite element modeling, hydrogen storage, and biomedical implants.

## Acknowledgements

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